

TOOELE CHEMICAL AGENT DISPOSAL FACILITY (TOCDF)



DEMONSTRATION TEST PLAN FOR THE AUTOCLAVE SYSTEM

Revision 0

EG&G DEFENSE MATERIALS, INC.

October 21, 2008

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FACILITY (TOCDF)**

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FOR THE
AUTOCLAVE SYSTEM**

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EXECUTIVE SUMMARY

The Tooele Chemical Agent Disposal Facility (TOCDF) was designed and built for the United States (U.S.) Army to destroy the chemical agent munitions stockpile at the Deseret Chemical Depot (DCD), located 20 miles south of Tooele, Utah. EG&G Defense Materials, Inc., (EG&G) operates the TOCDF under contract to the Army through the Chemical Materials Agency.

The U.S. Environmental Protection Agency (EPA) identification number for the TOCDF is UT5210090002. The facility operates under a Resource Conservation and Recovery Act (RCRA) Part B Permit, issued pursuant to the delegation of the State of Utah Department of Environmental Quality (DEQ), Division of Solid and Hazardous Waste (DSHW), under the Utah Administrative Code, Section 315 (R315). The TOCDF also operates under a Title V Air Permit administered by the DEQ, Division of Air Quality (DAQ). Under these permit requirements, the incinerator system must demonstrate the ability to effectively treat any hazardous wastes such that human health and the environment are protected.

The TOCDF secondary waste is generated as a result of demilitarization of the DCD chemical agent stockpile. Secondary wastes contaminated with chemical agent are treated on-site prior to shipment for final disposal at off-site facilities. The TOCDF proposes to use an autoclave to provide the on-site treatment to remove agent contamination from low-level-agent-contaminated wastes. Therefore, an Autoclave Demonstration Test (ADT) will be conducted to demonstrate treatment of secondary wastes in the autoclave by processing actual contaminated wastes to produce a treated residue that is not an agent vapor hazard [less than 1.0 Vapor Screening Limit (VSL)] or a contact hazard (< 1 mg/kg).

The test performance will be measured by quantifying the amount of agent removed from the wastes and the autoclave headspace agent concentration after treatment. The removal will be determined by spiking samples with Agent VX and then analyzing the spiked samples after treatment in the autoclave. A successful test will demonstrate removal of 99 % of the agent from the spiked samples and an agent concentration in the headspace of less than 1.0 VSL.

Materials that will be treated in the autoclave include chlorinated polymer materials, non-chlorinated polymer materials, wood, and miscellaneous debris. The ADT will establish feed limits for treating wastes at below incineration temperatures using four waste streams. The Demilitarization Protective Ensemble (DPE) waste stream will include DPE suits along with polyethylene sheeting and bags and possibly some DPE tape. The Life Support Systems (LSS) air hose waste stream will include LSS air hoses and the metal fittings. The wood waste stream will be wood wastes, plastics, and possibly metal banding straps. The fourth stream will be the plastic barrels that held the wastes or an additional waste stream. These four waste streams will be representative of the other wastes to be processed by the autoclave.

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LIST OF ACRONYMS AND ABBREVIATIONS

ACAMS	Automatic Continuous Air Monitoring System
ADAFc	ACAMS Dilution Air Flow Controller
ADT	Autoclave Demonstration Test
ASTM	ASTM International formerly American Society for Testing and Materials
AWFCO	Automatic Waste Feed Cutoff
CAL	Chemical Assessment Laboratory
CEMS	Continuous Emission Monitoring System
CFR	Code of Federal Regulations
CMA	Chemical Materials Agency
CPT	Comprehensive Performance Test
DAAMS	Depot Area Air Monitoring System
DAQ	State of Utah, Department of Environmental Quality, Division of Air Quality
DCD	Deseret Chemical Depot
DDAFc	DAAMS Dilution Air Flow Controller
DEQ	State of Utah, Department of Environmental Quality
DFS	Deactivation Furnace System
DPE	Demilitarization Protective Ensemble
DRE	Destruction and Removal Efficiency
DSHW	State of Utah, Department of Environmental Quality, Division of Solid and Hazardous Waste
DVS	Drum Ventilation System
EG&G	EG&G Defense Materials, Inc.
EPA	U.S. Environmental Protection Agency
ETL	Extreme Temperature Limit
GC	Gas Chromatography
GC/FPD	Gas Chromatograph/Flame Photometric Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
HAP	Hazardous Air Pollutant
HWMU	Hazardous Waste Management Unit
ICP/MS	Inductively Coupled Plasma/Mass Spectrometer
LIC	Liquid Incinerator
LOP	Laboratory Operating Procedure
LOQ	Limit of Quantitation
LSS	Life Support System
MACT	Maximum Achievable Control Technology
MDB	Munitions Demilitarization Building
MPF	Metal Parts Furnace

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

OPL	Operating Parameter Limit
PLC	Programmable Logic Controller
POHC	Principal Organic Hazardous Constituent
PPE	Personal Protective Equipment
PQL	Practical Quantitation Limit
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QP	Quality Plant Sample
R315	Utah Administrative Code Section 315
RCRA	Resource Conservation and Recovery Act
SDS	Spent Decontamination Solution System
SEL	Source Emission Limit
STEL	Short Term Exposure Limit
SVOC	Semi-Volatile Organic Compound
SW-846	Test Methods for Evaluating Solid Waste, 3rd Edition including Update III, USEPA, SW-846, December 1996.
TAP Gear	butyl rubber coated garments
TC	Ton Container
TE-LOP	Tooele Laboratory Operating Procedure
TOCDF	Tooele Chemical Agent Disposal Facility
TSDF	Treatment, Storage and Disposal Facility
U.S.	United States
VOC	Volatile Organic Compound
VSL	Vapor Screening Limit

LIST OF UNITS OF MEASURE

acfm	actual cubic feet per minute
Btu/hr	British thermal units per hour
Btu/lb	British thermal units per pound
Btu/ft ³	British thermal units per ft ³
cfm	cubic feet per minute
°C	degrees Centigrade
°F	degrees Fahrenheit
dscf	dry standard cubic feet
dscfm	dry standard cubic feet per minute
dscm	dry standard cubic meter
ft	feet
ft ³	cubic feet
g	grams
gpm	gallons per minute
hr	hours
hp	horsepower
inHg	inches of mercury
inWC	inches of water column
lb	pounds
lb/hr	pounds per hour
L/min	liters per minute
µg	micrograms
µL	microliter
m ³	cubic meters
mg	milligrams
mg/dscm	milligrams per dry standard cubic meter
min	minutes
mL	milliliters
mL/min	milliliters per minute
mm	millimeters
ng	nanograms
ppm	parts per million
ppmdv	parts per million on a dry volume basis
psig	pounds per square inch gauge
scfm	standard cubic feet per minute
Wt%	weight percent
ΔP	velocity pressure

LIST OF CHEMICAL SYMBOLS AND FORMULAS

Agent GB	Sarin or methylphosphonofluoridic acid 1-methylethyl ester or O-isopropyl methylphosphonofluoridate
Agent VX	O-ethyl-S-(2-diisopropylaminoethyl) methylphosphonothiolate
Al	aluminum
Ag	silver
As	arsenic
As ₂ O ₃	arsenic oxide
B	boron
Ba	barium
Be	beryllium
Cd	cadmium
CdO	cadmium oxide
Cl ⁻	chloride
Cl ₂	chlorine
CO ₂	carbon dioxide
CO	carbon monoxide
Co	cobalt
Cr	chromium
Cu	copper
EA-2192	S-(2-diisopropylaminoethyl) methylphosphonothioic acid
H	Levinstein mustard
HD	distilled mustard
HT	mixture <i>bis</i> (2-chloroethyl)sulfide and <i>bis</i> [2-(2-chloroethylthio)ethyl] ether
HF	hydrogen fluoride
HNO ₃	nitric acid
H ₃ PO ₄	phosphoric acid
Hg	mercury
HCl	hydrogen chloride
Mn	manganese
NaOH	sodium hydroxide
H ₂ SO ₄	sulfuric acid
mustard	<i>bis</i> (2-chloroethyl)sulfide
Ni	nickel
NO _x	nitrogen oxides
O ₂	oxygen

LIST OF CHEMICAL SYMBOLS AND FORMULAS (continued)

Pb	lead
PbO	lead oxide
PbCl	lead chloride
PCBs	polychlorinated biphenyls
PCDD	polychlorinated dibenzo-p-dioxin
PCDF	polychlorinated dibenzofurans
Q	1,2- <i>bis</i> (2-chloroethylthio)ethane
Sb	antimony
Se	selenium
SO ₂	sulfur dioxide
Sn	tin
T	<i>bis</i> [2-(2-chloroethylthio)ethyl]ether
TCDD	tetrachlorodibenzo-p-dioxins
Tl	thallium
V	vanadium
Zn	zinc

**LIST OF IDENTIFICATION CODES FOR AUTOCLAVE INSTRUMENTS
MONITORING REGULATED OPERATING PARAMETERS**

TT-241	Autoclave Bin 1 Temperature Thermocouple
TT-242	Autoclave Bin 2 Temperature Thermocouple
TT-243	Autoclave Bin 3 Temperature Thermocouple
TT-244	Autoclave Bin 4 Temperature Thermocouple
TT-201	Autoclave Headspace Temperature
PT-203	Autoclave Vacuum Pressure
TEN-083G *	Autoclave Agent GB Headspace Monitoring
TEN-083V *	Autoclave Agent VX Headspace Monitoring

* Only the Automatic Continuous Air Monitoring System (ACAMS) monitoring the agent contaminating the secondary waste will be on line during operation

1.0 INTRODUCTION

The Tooele Chemical Agent Disposal Facility (TOCDF) was designed and built for the United States (U.S.) Army to destroy the chemical agent munitions stockpile at the Deseret Chemical Depot (DCD). EG&G Defense Materials, Inc. (EG&G), operates the TOCDF under contract to the U.S. Army through the office of the Chemical Materials Agency (CMA). The U.S. Environmental Protection Agency (EPA) identification number for the TOCDF is UT5210090002. The facility operates under a Resource Conservation and Recovery Act (RCRA), Part B Permit issued pursuant to the delegation of the State of Utah, Department of Environmental Quality (DEQ), Division of Solid and Hazardous Waste (DSHW), under the Utah Administrative Code, Section 315. The TOCDF also operates under a Title V Air Quality Permit administered by the State of Utah, DEQ, Division of Air Quality (DAQ) and the Hazardous Waste Combustors Maximum Achievable Control Technology (MACT) rules. Under the requirements of these permits, the treatment systems must successfully demonstrate treatment of hazardous wastes while protecting human health and the environment.

The current TOCDF incinerators used to destroy the chemical weapons in the DCD stockpile are two Liquid Incinerators (LICs), a Deactivation Furnace System (DFS), and a Metal Parts Furnace (MPF). Secondary wastes are generated from the demilitarization of the DCD chemical stockpile. Secondary wastes contaminated with chemical agent are treated on-site prior to shipment for final disposal at off-site facilities. These secondary wastes include Personal Protective Equipment (PPE) such as Demilitarization Protective Ensemble (DPE) suits or butyl rubber coated garments (TAP Gear), packaging material (dunnage), plastic sheeting, spill absorbent, Life Support System (LSS) air hoses, spent activated carbon, and other miscellaneous wastes. Large amounts of wastes are currently being placed in storage, so the quantity of secondary wastes in permitted storage will continue to grow.

The current estimate of secondary wastes generated by TOCDF operations is 1,000 tons. The Mustard Campaign requires the munitions to be processed in the MPF; therefore, only a limited time is available for the processing of secondary wastes in the MPF. This will result in significantly extending TOCDF closure to allow processing of both the munitions and the secondary wastes in the MPF. To minimize this impact, TOCDF intends to install and operate an autoclave for the purpose of treating secondary waste with an Automatic Continuous Air Monitoring System (ACAMS) reading greater than or equal to (\geq) 1.0 Vapor Screening Limit (VSL).

Secondary wastes will be sorted based on the waste evaluation conducted in the Drum Ventilation System (DVS). Those wastes with an ACAMS reading that is < 1.0 VSL will be shipped offsite to a Subtitle C Treatment, Storage, and Disposal Facility (TSDF). Those wastes with an ACAMS agent concentration ≥ 1.0 VSL are candidates for treatment in the autoclave.

An autoclave is a sealed vessel into which pressurized steam is injected. The elevated pressure at which the autoclave is operated allows the steam to reach temperatures > 212 °F, the boiling point of water. These increased temperatures and steam from the autoclave will result in the hydrolysis of the chemical agents to non-toxic compounds that result in waste decontamination without waste incineration. Then these decontaminated wastes can be shipped off-site to a licensed Subtitle C TSDF for disposal as hazardous wastes.

A test is proposed to demonstrate the performance of this miscellaneous treatment unit. This test will be referred to as the Autoclave Demonstration Test (ADT). A set of samples will be collected to characterize the autoclave effluent streams, and a set of process data will also be collected to document the performance of the autoclave under the test conditions. Spiked samples will be recovered and analyzed to document the treatment effectiveness of the autoclave; and agent emissions will be monitored during the ADT.

EG&G will conduct the ADT, and collect the effluent stream samples. At the conclusion of the process cycle, the headspace above the treated wastes will be monitored for agent. The only process samples to be collected will be the condensate samples from the condensers that cool the exhaust from the autoclave and the spiked samples. These condensate samples will be analyzed by the TOCDF Chemical Assessment Laboratory (CAL) and a subcontractor laboratory. Spiked samples will be recovered and analyzed for agent to determine how much spiking agent remains after treatment. The exhaust gas sampling and analytical methods to be used to quantify specific ADT parameters are from SW-846 (1) and Tooele Laboratory Operating Procedures (TE-LOPs).

1.1 BACKGROUND CHEMISTRY DISCUSSION

Autoclave processing was selected for secondary wastes because the environment it creates causes the chemical agents to undergo a hydrolysis chemical reaction. This reaction causes a substituent element in the compound to be replaced by a hydroxide group (-OH). In the case of mustard, the chlorine (the substituent element) is replaced with the hydroxide group, producing thiodiglycol. However, this reaction proceeds at different rates for different compounds, and some compounds do not undergo hydrolysis reactions. An example of this is the different forms of Lewisite: Lewisite L1 and Lewisite L2 are easily hydrolyzed, but Lewisite L3 does not hydrolyze because it does not have a chlorine atom available for the hydroxide group to replace.

Nerve agents GB and VX are compounds that can undergo hydrolysis reactions. They are both phosphonate esters that undergo substitution reactions at the phosphorus to yield substituted compounds that are much lower in toxicity than the original compounds. Agent GB will undergo hydrolysis by the hydroxide group replacing the fluorine atom to yield isopropyl methylphosphonic acid. Agent VX can undergo two different substitution reactions and yield either ethyl methylphosphonic acid or S-(2-diisopropylaminoethyl) methylphosphonothioic acid (EA-2192). The reaction products are dictated by the reaction conditions.

Table 1-1 shows the relative reaction rates for the hydrolysis reactions in aqueous solutions for the chemical agents treated at TOCDF based on data from the literature (2, 3, 4). This table shows the relative reaction rates as measured by the hydrolysis rate data and the thermal decomposition rate. The autoclave will operate at elevated temperatures and pressures, which will increase the reaction rate for the hydrolysis reaction.

TABLE 1-1. RELATIVE REACTION RATES FOR CHEMICAL AGENTS

Agent	Vapor Pressure		Volatility		Hydrolysis Rate (Half Life)	Thermal Decomposition Rate (Half Life)
	@ 0 °C (torr)	@25 °C (torr)	@ 0 °C (mg/m ³)	@25 °C (mg/m ³)		
VX	4.20E-05	8.78E-04	0.662	12.6	40 days @ 25 °C & pH = 7	35 hours @ 150 °C
GB	0.41	2.48	3,370	18,700	47 hours @ 25 °C & pH = 6	2.5 hours @ 150 °C
HD		0.106		906	8.5 minutes @ 25 °C & pH = 7	Decomposes @ 149 - 177 °C

An evaluation of the data in Table 1-1 and a review of literature on the hydrolysis of chemical agents (2, 3, 4) indicated that the chemical agents present in the secondary wastes could be successfully decontaminated in an autoclave. Therefore, two series of tests were performed to help define the operating parameters for a commercial sized autoclave to treat secondary wastes. The first test was conducted at the Bondtech factory in North Carolina (5). This series of tests were conducted in a 3-ft diameter autoclave with a surrogate compound in a wide variety of matrices. This test series showed that the surrogate could be driven from the test matrices, but the compound did not undergo hydrolysis in the gas phase. This would indicate the approach was sound, but the thesis would need to be tested with the actual chemical agents to determine if the project could be successful.

The next test series was begun using Agent VX since it was viewed to be the hardest agent to hydrolyze. The preliminary data has shown that > 99 % of the agent was destroyed or removed from the spiked sample, but agent concentrations were found in the aqueous streams from the treatment process. The tests using wood showed that a lower amount of agent was removed from the spiking matrix, but lower concentrations remained in the headspace gas and the aqueous effluents. The other information developed by the second series of tests was the absence of EA-2192 in the matrices spiked and the effluent streams from the autoclave (6).

1.2 AUTOCLAVE DEMONSTATION TEST PLAN ORGANIZATION

This plan describes the operating conditions, samples collected, and sample analyses for the ADT. The Quality Assurance Project Plan (QAPP) (see Appendix A) describes the sampling and analyses to be conducted. Appendix B provides the Shakedown Plan that will be used to prepare the autoclave for the ADT. Appendix C provides additional supplemental information.

1.3 FACILITY INFORMATION

The TOCDF is located in EPA Region 8, and the TOCDF EPA Identification Number is UT5210090002, which is also the DSHW RCRA Permit number. The DCD Title V Operating Permit Number is 4500071001.

The ADT points of contact are:

Thaddeus A. Ryba, Jr., TOCDF Site Project Manager
11620 Stark Road
Stockton, UT 84071
(435) 833-7439

Mr. Gary McCloskey, TOCDF General Manager
EG&G Defense Materials, Inc.
11600 Stark Road
Stockton, UT 84071
(435) 882-5803

Mr. Steven Brow, ADT Test Director
EG&G Defense Materials, Inc.
11600 Stark Road
Stockton, UT 84071
(435) 882-5803

1.4 SECONDARY WASTE SORTING PROCESSES

The TOCDF secondary wastes are generated from the demilitarization of chemical munitions and bulk containers. They are typically single-use items, discarded process equipment, or agent-contaminated dunnage. These secondary wastes were generated within the TOCDF Munitions Demilitarization Building (MDB) and were exposed to liquid chemical agent or chemical agent vapors within the MDB. Therefore, a high probability exists that secondary wastes are contaminated with chemical agent, which requires on-site treatment.

The secondary waste will be sorted and treated in the autoclave, if necessary. Containerized secondary waste will initially be moved from its current storage location in Area 10 to Igloo 1633, which was permitted as a Container Storage Hazardous Waste Management Unit (HWMU). Igloo 1633 will serve as a staging area to hold drums of secondary waste that are to be sorted, and sorted drums of waste that are being held to accumulate a sufficient quantity to support autoclave operations.

Drums of secondary waste are sorted into two waste categories at the DVS. Each waste drum is placed into the DVS, where the headspace of the drum is monitored for chemical agent using an ACAMS. Secondary wastes with headspace ACAMS results that are ≥ 1.0 VSL are candidates for additional on-site treatment in either the autoclave or in the MPF.

1.5 AUTOCLAVE SECONDARY WASTE MANAGEMENT SUMMARY

Drums of secondary waste with ACAMS results of ≥ 1.0 VSL will be moved to Igloo 1631 for autoclave treatment. The autoclave will be installed in Igloo 1631, which is located in DCD Area 10. This igloo was previously used to sample the DCD Mustard Ton Container (TC) Stockpile. The gloveboxes that were used to sample the TCs have since been removed in anticipation of the autoclave installation. The installation includes:

- an autoclave,
- the boiler to provide steam to the autoclave and vacuum systems,
- chillers to cool the gas stream and condense water vapor,
- an air blower that supplies cooling air to the autoclave after the thermal treatment,
- a holding tank to collect the condensate until it can be pumped to storage,
- a Programmable Logic Controller (PLC), and
- two 1,000-gallon Spent Decontamination Solution System (SDS) Tanks to store aqueous wastes until shipment offsite.

Waste is fed to the autoclave in 64.7-cubic-foot (ft³) waste bins (waste may be processed in the storage drums provided adequate treatment data can be developed). The autoclave can hold up to four waste bins. A liner is placed in the bin prior to transferring the content of multiple drums of secondary waste into the bin. The transfer of wastes from the original storage containers to the waste bins is done using mechanical assistance and helps decompress the waste from the drums. The filling of the waste bins and the opening of the original storage containers occurs

next to a fume hood, which is located on one side of the autoclave vessel to allow the air flow from the worker side, across the waste, and into the fume hood. Exhaust gas from the fume hood is ducted to the carbon filter system, which supports autoclave and DVS operations that will occur concurrently in adjacent Igloo 1632. The autoclave load/unload door is hinged on the side opposite the fume hood.

A thermocouple inside a thermocouple well is inserted into the wastes loaded in each filled waste bin that occupies one of the four possible positions in the autoclave. These thermocouples will monitor the temperature of the wastes during the treatment process.

Once loaded, the door to the autoclave is sealed closed. A steam eductor draws a vacuum on the autoclave interior down to 10 inches of mercury (inHg). The air and volatilized agent drawn from the autoclave and the eductor steam are mixed at the eductor resulting in a water-saturated gas. This gas passes through two condensers that remove the moisture from the gas stream before the gas exhausts to a carbon filter system. The condensed water vapor is drained to a 175-gallon holding tank. When the holding tank contents reach about 140 gallons, it will be pumped to one of two 1,000-gallon SDS tanks.

After the autoclave pressure reaches 10 inHg, the steam eductor is stopped, and steam is directed from the boiler to the autoclave. The autoclave is a direct-steam design, so the steam injected into the autoclave contacts the secondary waste material inside (as opposed to an indirect-steam design where the steam passes through tubes that run through the autoclave). The maximum operating pressure and temperature of the autoclave is 85 pounds per square inch gauge (psig) and 330 °F, respectively.

Autoclave operations are controlled by a dedicated PLC. The operator can select and control the operating temperature and duration of temperature soak. Once the temperatures inside the waste bins, as measured by the thermocouples, reach the setpoint, a timer starts with a predetermined time, and the temperature and pressure within the autoclave is maintained for that time period.

The process temperature soak time will be divided into two segments to facilitate the movement of steam throughout the waste load. The first segment of the soak time will melt the plastic bags and open the wastes to treatment of the agent. The first segment will be followed by an evacuation of the autoclave to about 10 inHg, which is followed by steam treatment to again reach the minimum temperature at all of the thermocouples. When the minimum temperature is reached, the soak timer will start the second segment of the temperature soak time.

When the process timer expires, steam is once again directed to the eductor, and the interior of the autoclave is evacuated to a vacuum of 10 inHg. This post-treatment evacuation step is intended to dry and cool the treated waste inside the autoclave. The water vapor removed from the autoclave is again condensed and collected in the holding tank, which will be emptied at the end of the run.

Upon completion of the third evacuation process step, the interior of the autoclave is backfilled with ambient air at atmospheric pressure. Ambient air is passed through the autoclave using an external blower introducing about 300 cubic feet/minute (cfm) to dry and cool the wastes. The air within the autoclave is then monitored for agent concentrations. If the ACAMS results show that the agent concentration is < 1.0 VSL, the treatment of the secondary waste to remove agent contamination is complete, and the treated wastes are removed from the autoclave, and shipped to a Subtitle C TSDF. If the ACAMS reading is ≥ 1.0 VSL, then the waste is treated by another autoclave cycle.

The liner that was initially placed in the waste bins will be sealed to enclose the secondary wastes. The full bin is taken outside the igloo, and the bin's contents are transferred into a roll-off, which will be filled until the weight limit or volume limit are reached. The roll-off will then be covered and shipped off-site to a Subtitle C TSDF as hazardous waste.

Note that the temperature at which the autoclave is operated does not result in a reduction in the volume of the waste treated. In other words, autoclave operations do not incinerate the wastes it treats, and therefore, does not result in the generation of ash. The polyethylene bags (in which secondary waste is typically packed) melt, exposing the wastes inside to the steam, which hydrolyzes the agent compounds into non-toxic compounds.

In addition, the wastes treated in the autoclave are classified as hazardous waste only because they are residues from the treatment of chemical agent contaminated items. In general, the secondary wastes (e.g., plastic or wood) do not include chemical compounds that would categorize the items as hazardous waste when discarded.

1.6 AUTOCLAVE DEMONSTATION TEST OBJECTIVES

The objectives for the TOCDF ADT are to:

- Demonstrate the ability of the autoclave to treat a variety of secondary wastes to agent concentrations that allow the safe handling of the wastes until they can be shipped to a Subtitle C TSDF. The demonstration that the agent has been destroyed will be based on spiked samples and the amount of agent remaining on the spiked samples after treatment.
- Establish operating conditions governing the treatment of secondary wastes in the autoclave that establish an operating envelope that allows the treatment of the secondary wastes based on waste temperatures and treatment times.
- Characterize the condensate samples to include Agent VX, agent hydrolysis compounds, Volatile Organic Compounds (VOCs), Semi-Volatile Organic Compounds (SVOCs) and metals, to establish the final treatment for the condensate waste stream.
- Monitor for agent in the effluent streams to determine where the agent is treated.

1.7 PROPOSED ADT PROGRAM

This ADT will establish permit limitations for Agents GB, VX, and mustard contaminated secondary wastes. The ADT will demonstrate treatment of a secondary waste feedstock to demonstrate worst-case agent contamination for secondary waste treatment. Since the autoclave is a batch feed treatment system, the shakedown period will be used to determine the optimum charge volume and charge interval for secondary wastes. The system pressures will vary between a vacuum of about 10.0 inHg to less than 85 psi under steam treatment. Operating Parameter Limits (OPLs) for the autoclave will be established by the ADT.

Agent concentrations in the autoclave headspace will be monitored using ACAMS, and a Depot Area Air Monitoring System (DAAMS) will be used to monitor Agent VX in the duct between the condensers and the carbon filters. The headspace of the autoclave will be monitored by an ACAMS after treatment is completed, but an agent concentration in the waste will not be determined; therefore, a DRE based on agent emissions cannot be calculated. However, the removal of agent from the spiked samples will be measured to demonstrate successful treatment. The success of the test is based both on the headspace monitoring and the demonstration of agent removal from the spiked samples.

1.8 ADT SAMPLING AND ANALYTICAL PROTOCOLS

Discussions of the sampling and analysis procedures are provided in Appendix A. The structure of the ADT is designed to meet the objectives in Section 1.6. The exhaust gas sampling and analytical methods to be used to quantify specific ADT parameters are taken from SW-846 (1) and TE-LOP. These methods are described by the following:

- The DAAMS will monitor the exhaust gas in the duct between the steam inductor and the common duct to the carbon filters (TE-LOP-522). The DAAMS tubes will be analyzed for Agent VX (TE-LOP-562).
- The ACAMS will monitor the headspace in the autoclave to determine whether Agent VX remains unhydrolyzed in the wastes (TE-LOP-524).
- Spiked samples will be analyzed after treatment to demonstrate that at least 99 % of the agent has been removed from the spiked samples. The spiked samples will be extracted and then analyzed (TE-LOP-572).
- Condensate samples will be analyzed for Agent VX and its hydrolysis products by the CAL. A subcontractor laboratory will analyze the condensate samples for VOCs using SW-846, Method 8260; for SVOCs using SW-846, Method 8270; and for metals using SW-846, Method 6020 (1).

1.9 FINAL WASTE FEED LIMITS

Four types of secondary wastes will be demonstrated during the ADT, but other secondary wastes will be qualified for treatment in the autoclave based on the results of this test. The four bins will be filled with four different types of secondary waste. One bin will be filled with DPE suits, a second with LSS hoses, a third with wood, and the fourth with pieces of plastic drums. Any secondary wastes containing sludges will only be treated in the MPF. Although not all secondary waste types will be demonstrated during the ADT, each will be approved for processing in the autoclave as a result of this ADT, with the exception of carbon and contaminated soils, which will be demonstrated in separate demonstration tests.

2.0 ENGINEERING DESCRIPTION OF THE AUTOCLAVE TREATMENT SYSTEM

The autoclave will be installed in Igloo 1631, which is located in DCD Area 10. This igloo was previously used to sample the DCD Mustard TC Stockpile. The gloveboxes that were used to sample the TCs have since been removed in anticipation of the autoclave installation. The entire installation includes:

- A direct steam autoclave;
- A boiler and associated water treatment system;
- A conveyor system to load the bins into the autoclave;
- A vacuum system;
- A two stage condensing system;
- Air blower to cool and dry autoclave contents;
- One 175-gallon holding tank;
- A PLC; and
- Two 1,000-gallon SDS Tanks.

2.1 AUTOCLAVE DESCRIPTION

The autoclave is 6 feet in diameter and 20 feet long with a capacity to hold four waste bins or 16 drums. The waste bins are made from stainless steel and they are 4 feet 6 inches wide by 4 feet 8 inches deep by 3 feet 1 inch tall. The autoclave is a direct steam autoclave, which means that the steam comes into direct contact with the waste in the autoclave and does not provide heat to tubes located inside the autoclave. The maximum pressure to the autoclave is 85 psig and the maximum temperature is 330 °F.

A fume hood is attached along the side of the opening to the autoclave to provide engineering controls over any emissions that might be generated by transfer of wastes to the bins.

The autoclave is equipped with a powered lift to move the bins onto a chain drive that moves the bins to the inside of the autoclave. The side-hinged door on the autoclave can be closed and an air-tight seal established to allow the development of a vacuum inside the autoclave.

Temperatures in the autoclave are controlled using thermocouples placed inside thermocouple wells that allow insertion into the waste load to record the minimum temperature experienced by the wastes. These temperatures ensure the wastes have reached the treatment temperature by the PLC controlling steam. Steam is added to the autoclave until all the thermocouples reach the minimum temperature. The temperature is maintained at the setpoint by the addition of steam to the autoclave until the minimum time at temperature is reached for the waste load.

Any moisture that condenses on the inside of the autoclave is collected in drains in the floor of the autoclave, and the condensate is drained, sent to the steam eductor, condensers, and to a holding tank before it is pumped to the SDS tanks. The steam vent on the autoclave is directed to the steam eductor followed by a two-stage cooler to condense the water vapor and any organic compounds exiting the system.

2.2 BOILER DESCRIPTION

The boiler is rated at 5.2-million British thermal units per hour (Btu/hr), which is capable of operating to 125 psig. A water treatment system is also included to provide treated water to the boiler. A water softener is used to treat water in the boiler to minimize fouling of the boiler. The water treatment will also remove oxygen from the water to reduce boiler corrosion. The boiler provides steam to the autoclave for heating the waste and steam to operate the steam eductor which provides the vacuum to lower the pressure in the autoclave. The boiler is fired by natural gas.

2.3 VACUUM SYSTEM DESCRIPTION

The steam eductor was designed to evacuate the autoclave to a pressure of approximately 5.0 inHg in four minutes. The evacuation time may be influenced by the amount of water or organic compounds that off-gas during the evacuation period. The steam that operates the eductor will be condensed in the cooling system and collected in the SDS tanks. Use of a steam eductor saturates the air stream that is removed from the autoclave with water.

2.4 MOISTURE CONDENSING SYSTEM DESCRIPTION

The cooling system is a two-stage chiller. The first stage is a propylene glycol-chilled condenser with a cooling tower. The gases removed from the autoclave are initially cooled in the heat exchanger after going through the steam eductor. The autoclave gases are cooled to ambient temperatures with this first stage of cooling.

The second stage of cooling is a refrigerant-cooled condenser used to further cool the gases removed from the autoclave. Both condensers are used to remove the moisture in the gases exiting the autoclave during evacuation. Once the moisture is removed by chilling the gases, the chilled and dried air stream is directed to an existing activated carbon filter system, which is used to remove any agent vapors prior to the air being released to the environment. The carbon filter system services both the autoclave operations in Igloo 1631 and the DVS operations in Igloo 1632.

2.5 AIR BLOWER SYSTEM

An air blower system was added to the autoclave system to provide air to cool and dry the wastes prior to opening the autoclave door to remove the wastes. The blower was designed to flow approximately 300 scfm room air across the load. The air exits the autoclave through the steam vent on the autoclave and is directed to a two-stage condenser to condense the water vapor and any organic compounds exiting the system. The condensate from the cooling step is drained to the condensate holding tank prior to being pumped to the SDS tanks.

2.6 COMPUTER CONTROL SYSTEM

The autoclave system is controlled with a PLC that automates the autoclave treatment process, evaluates the status, and records selected process parameters during treatment. Temperatures in the wastes will be monitored by the PLC until the minimum temperature is reached by each thermocouple. Once the temperature set point is reached, the PLC will start a timer for the minimum time for waste treatment. Temperatures and pressures will be recorded with a time line to allow reconstruction of the processing of each batch in the autoclave. The PLC will have the capability to download its stored information to a computer for archiving data and preparation of the daily report.

2.7 SDS TANK STORAGE DESCRIPTION

The water condensed from the autoclave is collected and placed in two 1,000-gallon storage tanks located inside the igloo. These tanks will be operated as 90-day accumulation tanks to hold the collected autoclave condensate. Condensate will be collected from the cooling system, pumped to the SDS tanks by the condensate pumps, and stored in the tanks until the contents can be analyzed and transferred to a Subtitle C TSDF.

2.8 AWFCO DESCRIPTION

There are no Automatic Waste Feed Cutoffs (AWFCOs) associated with the autoclave. The operation of the autoclave will be controlled by the operations SOPs, which will be used to control the start of the autoclave cycle to ensure the SDS tanks are not overfilled or the door is not opened unless the ACAMS readings are < 1.0 VSL.

2.9 AGENT MONITORING SYSTEMS

Operations of the agent monitoring systems are discussed in Attachment 22 to the TOCDF RCRA Permit (8) and in Appendix A of this document. Agent concentrations in the autoclave and in the exhaust gases are monitored using ACAMS and DAAMS. Agent monitoring during the ADT will include Agent VX. These systems have undergone extensive testing and evaluation under both simulated and actual field conditions.

Operations of the ACAMS and DAAMS are controlled by Laboratory Operating Procedures. These systems use a Gas Chromatograph with a Flame Photometric Detector (GC/FPD) for the detection of Agent VX. Agent VX is not volatile enough to be easily analyzed by GC, so a silver fluoride impregnated conversion pad is needed to convert Agent VX to its more volatile G-Analog. The G-Analog can be easily analyzed by GC. The silver fluoride pad will be located at the distal end of the sample probes for both the ACAMS and DAAMS. These pads will be changed as directed by Attachment 22 (8).

The precision and accuracy of each monitoring system is determined through actual on-site testing after installation of the equipment and then checked at periodic intervals. These data are used to establish quality control bounds, calibration and challenge frequencies, and procedures. These challenge frequencies and procedures are then delineated in a quality control plan for each system.

The ACAMS will monitor the autoclave headspace after completion of the autoclave cycle and will run for a minimum of two cycles on the headspace gas and if both samples are < 1.0 VSL, then the wastes will be released to be shipped off-site. If the agent concentration is ≥ 1.0 VSL, then the contents will be treated through another cycle in the autoclave.

3.0 SAMPLING AND ANALYSES PROCEDURES

The sampling and analysis objectives for the TOCDF ADT are to:

- Demonstrate control of agent emissions by showing that the agent concentrations after treatment remain below the detection limits in the headspace of the autoclave.
- Demonstrate removal of agent by analyzing spiked samples after treatment.
- Determine whether other organic compounds are off-gassed during autoclave treatment by analyzing the condensate samples for VOCs and SVOCs.

3.1 SAMPLING LOCATIONS

Three types of samples will be collected for the ADT: exhaust gas samples, condensate samples and spiked samples. The exhaust gas samples will be collected in the autoclave headspace to ensure there is no remaining agent after treatment of the wastes and in the duct between the steam eductor and the activated carbon filter beds. Condensate samples will be collected from the discharge side of the condensate transfer pump between the condensate holding tank and the SDS tanks. The spiked samples will be recovered from the wastes in the bins using a string from the sample to the surface of the wastes.

3.2 SAMPLING METHODS

Autoclave gases will be monitored using the ACAMS and DAAMS. As a safety precaution, autoclave headspace gases will be monitored for Agent VX with an ACAMS using TE-LOP-524 to ensure that the Agent VX concentration is < 1.0 VSL. The gases leaving the autoclave through the steam valve will be sampled by DAAMS to determine the amount of agent leaving in those gases using TE-LOP-526. These sampling methods are discussed in Appendix A (QAPP).

Liquid effluent from the autoclave will be sampled to determine the mechanism for decontamination of the wastes in the autoclave. The liquid effluent will be collected in the condensate holding tank until there is enough to allow pumping to the SDS tanks for storage. A set of samples will be collected using a tap on the discharge side of the pump as the holding tank is emptied. The samples will be collected as directed by ASTM D 3370-95a (9) as discussed in Appendix A (QAPP).

3.3 ANALYSES METHODS

The DAAMS samples are analyzed by the CAL and the liquid effluent samples are analyzed by the CAL and the subcontract laboratory.

3.3.1 Gas Sample Analyses Methods

The ACAMS and DAAMS methods are discussed in Appendix A (QAPP). The ACAMS analyses method is part of TE-LOP-524 because the analysis module is part of the ACAMS instrument. The gas sample is collected on the absorbent and then the absorbent is thermally desorbed into a field GC/FPD that conducts the actual analysis. The GC separates the agent from the other compounds and the FPD detects the phosphorus when the agent burns.

The DAAMS tubes are analyzed according to TE-LOP-562. The tubes are thermally desorbed into a GC/FPD where the organic compounds are separated from the agent by the GC and the agent is detected by the light emitted from the phosphorus in the agent when it burns in the FPD. The concentration is calculated by comparison to external standards.

3.3.2 Liquid Samples Analyses Methods

The liquid samples are analyzed for agent and agent decomposition products in an effort to better understand the decontamination process for wastes in the autoclave. The organic compounds will be extracted from the water matrix using chloroform and then analyzed by GC/FPD to detect the agent present in the samples.

The TOCDF CAL will analyze the agent decomposition products in the condensate samples. The compounds will be extracted from the aqueous sample using solid phase extraction device in accordance with SW-846, Method 3535A. The compounds will be stripped from the extraction device using a solvent that is compatible with the analyses method such as LC/MS for the determination of the decomposition products.

The condensate samples will also be analyzed by the subcontract laboratory for VOCs by Gas Chromatograph/Mass Spectrometer (GC/MS) using SW-846, Method 8260; for SVOCs by GC/MS using SW-846, Method 8270; and for metals by Inductively Coupled Plasma/Mass Spectrometer (ICP/MS) using SW-846, Method 6020.

3.3.3 Agent Spiked Samples

The spiked samples were sized to allow the entire sample to be extracted for analyses. A 10-gram (or smaller) sample matrix was used in all cases to allow the agent to be spiked on the matrix, treated, recovered from the waste, extracted, and the extract analyzed. Method TE-LOP-572 will be used for the extraction and analyses of the samples.

4.0 AUTOCLAVE DEMONSTRATION TEST SCHEDULE

The ADT is scheduled for the second quarter of 2009. The DSHW will be notified at least 30 days in advance of the actual ADT date. The ADT will begin after TOCDF has: received approval of the ADT Plan; successfully completed autoclave installation and startup; and successfully completed the autoclave shakedown. The ADT should span about 5 days: 1 day for setup, 3 days of testing, and 1 day for cleanup. Typical test runs will be one complete cycle of the autoclave.

The ADT will consist of one test condition with three replicate sampling runs. One run per day is planned. Actual sampling time during each sampling run will last about 8 hours. The autoclave will be charged with the wastes to be decontaminated and the process started. Samples of the exhaust gases will be collected during the initial venting of the autoclave to determine if the agent is removed from the wastes at this point. Exhaust gas sampling will begin again when the autoclave is vented after the first segment of steam treatment of the wastes. Once the autoclave is vented after the final segment of steam treatment, the autoclave will be filled with ambient air, which will flow through the autoclave for a minimum of two hours. The flow will then be stopped to allow any agent present to reach an equilibrium concentration in the headspace over the wastes. The headspace will then be analyzed for agent using the ACAMS. This time period will be set based on the test results used to establish the operating conditions for the autoclave.

5.0 AUTOCLAVE DEMONSTRATION TEST PROTOCOLS

The ADT will consist of three replicate runs performed with a mixture of wastes. The wastes used for this test will be spiked with Agent VX to demonstrate that the autoclave is capable of destroying the hardest agent. The following subsections will discuss the waste to be treated, the test operating conditions, waste feed rates, and total waste to be processed.

5.1 WASTE CHARACTERIZATION

The State of Utah has defined chemical agents as acutely hazardous and identified them as a P999 waste. The same identification is applied to any waste that has been contaminated by chemical agents. Once the wastes have undergone successful treatment, the waste code is changed to F999. In special cases where it is not possible to analyze the treated residues, the wastes will carry a special waste code of P999/F999.

Several waste streams are proposed for treatment in the autoclave, but only four will be demonstrated by the ADT: DPE suits, LSS hoses, wood, and polyethylene/ polypropylene drums. Table 5-1 shows the secondary waste inventory for Agent GB and Table 5-2 shows the Agent VX secondary waste inventory summary. These tables demonstrate that the major items are directly covered by wastes treated in the ADT.

The wastes treated in the ADT will be more than just those listed in Tables 5-1 and 5-2. Wastes designated as DPE suits will be DPE suits and the plastic bags the suits were stored in with possibly some DPE tape. Wastes designated as wood may also contain items such as metal banding, plastic, paper, and tape. The wastes designated as LSS air hoses are anticipated to be just hoses, but may contain a small fraction of other debris. The polyethylene/polypropylene drums will be cut into pieces to accommodate fitting the drums into the fourth bin, which will include the drum lids and metal closures for the drums. The materials will not be sorted to more closely resemble the actual wastes that will be treated on a daily basis.

After treatment in the autoclave, the wastes can be shipped off-site to a Subtitle C TSDF. Activated carbon treatment will be demonstrated in a separate demonstration test once the analytical methods are developed to show that the treatment of carbon hydrolyzes the agent present even if it is absorbed on activated carbon.

One waste category listed in Table 5-2 as a mixture is air lock trash. In an effort to better understand the waste, two drums of airlock trash were opened and the contents sorted; the summary is shown in Table 5-3. There were four items found in the drums: DPE booties, butyl gloves, DPE tape, and plastic bags. Therefore, airlock trash can be treated in the autoclave based on the major items demonstrated as ADT wastes.

TABLE 5-1. AGENT GB SECONDARY WASTE INVENTORY

Profile	lb	Percent of Total	Planned Treatment	Autoclave Treatment Covered by
Carbon	106,740	29.2	Autoclave	Separate Carbon Demo Test
Wood Dunnage	91,032	24.9	Autoclave	ADT Demo - Wood
Trash & Plastic	53,407	14.6	Autoclave	ADT Demo - Combined Wastes
Agent Contaminated Debris	70,706	19.3	Autoclave	ADT Demo - Combined Wastes
Other	43,948	12.0	Autoclave	ADT Demo - Combined Wastes
Totals	365,833	100.0		

TABLE 5-2. AGENT VX SECONDARY WASTE INVENTORY

Profile	Weight (lb)	Percent of Total	No. of Containers	Planned Disposition	Autoclave Treatment Covered by
Paint Waste	7,527	1.3	87	MPF	Not Applicable
DPE Suits	200,398	33.5	1,569	Autoclave	ADT Demo - DPE Suits
Plastic	37,728	6.3	544	Autoclave	ADT Demo - DPE Suits
Charcoal Filters	7,136	1.2	55	Autoclave	Separate Carbon Demo Test
Absorbent, Cardboard	6,506	1.1	60	MPF	ADT Demo - Wood
Metal Maint. Equip	36,089	6.0	236	MPF	Not Applicable
TAP Gear	136,665	22.8	1,119	Autoclave	ADT Demo - Combined Wastes
Wood Dunnage	18,966	3.2	149	Autoclave	ADT Demo - Wood
Airlock Trash, Booties	76,715	12.8	592	Autoclave	ADT Demo - Combined Wastes
TYVEK, SARANEX	3,047	0.5	50	Autoclave	ADT Demo - DPE Suits
Mine Debris	12,429	2.1	305	Autoclave	ADT Demo - Combined Wastes
CAL Lab Prefilters	3,216	0.5	32	MPF	Not Applicable
SDS Strainers/Debris	1,528	0.3	12	MPF	Not Applicable
LSS Air Hoses	23,264	3.9	199	Autoclave	ADT Demo - LSS hoses
V/G Pads	3,881	0.6	64	MPF	Not Applicable
Cotton Goods	575	0.1	6	Autoclave	ADT Demo - Wood
Spent Sodium Lamps	607	0.1	10	Autoclave	ADT Demo - Combined Wastes
SDS Tank Liners	2,949	0.5	28	MPF	Not Applicable
SDS Sludge	2,315	0.4	13	MPF	Not Applicable
Non-Burnable Maint.	15,111	2.5	131	MPF	Not Applicable
SCBA Assembly	153	0.03	2	MPF	Not Applicable
TOX Sump Sludge	1,806	0.3	17	MPF	Not Applicable
Totals	598,611	100.0	5,280		

TABLE 5-3. AIRLOCK TRASH CHARACTERIZATION

Parameter	Drum 1 Contents		Drum 2 Contents	
	(lb)	(Wt%)	(lb)	(Wt%)
Total Contents Weight	111		76	
DPE Booties	57	51.4	22	28.9
Butyl Gloves	25	22.5	22	28.9
DPE Tape	17	15.3	14	18.4
Plastic bags	12	10.8	18	23.7
		100.0		100.0

The wastes will not be characterized in detail for this test, but the agent concentration in the headspace will be monitored after treatment as a demonstration that chemical agents are not present in the autoclave and it safe to remove the wastes from the autoclave. An ACAMS will be used to monitor the agent concentrations in the headspace of the autoclave as the characterization of the waste stream.

5.2 AGENT DESTRUCTION DEMONSTRATION

Materials designated as secondary wastes are mainly commercial items contaminated by contact with agent vapors or by surface exposure to liquid agents. These items are generally surface decontaminated with bleach or caustic before being placed in storage, which would further break down the agent concentration while in storage.

TOCDF will demonstrate the removal of Agent VX from a series of spiked samples as a demonstration that the wastes treated in the autoclave are safe to ship to a Subtitle C TSDF. The spiked samples will be placed in each waste bin and distributed within the bins to ensure all the wastes are treated by steam and temperature. After treatment, the spiked samples will be recovered from the wastes and analyzed for Agent VX. TOCDF will show that the Agent VX is not a vapor hazard by monitoring the autoclave headspace and show that the Agent VX concentration is < 1.0 VSL. The analysis of the spiked samples will show that the minimum amount of Agent VX removed is 99 % and therefore, the waste is not a contact hazard because the agent VX concentration will be ≤ 1 mg/kg based on the 10-gram samples.

5.3 TREATMENT STRATEGY FOR NON-ADT WASTE MATRICES

Other secondary waste matrices not demonstrated during the ADT will be processed by limiting their feed based on their similarity to wastes demonstrated in the ADT. Tables 5-1 and 5-2 list the different secondary wastes and their justification for treatment in the autoclave without demonstrating every specific waste matrix. The treatment of DPE suits will demonstrate that the autoclave can decontaminate plastics at the autoclave operating conditions. LSS hoses will demonstrate the treatment of rubber products and cotton products. The DPE suits and LSS air hoses will demonstrate the treatment of TAP gear. The treatment of DPE suits and LSS air hoses will demonstrate the treatment of airlock trash. Wood will demonstrate that the agent can be hydrolyzed when it is absorbed into a porous material such as wood. Absorbent packages soaked in chemical agents will not be processed in the autoclave, but only in the MPF.

5.4 PROPOSED ADT OPERATING CONDITIONS

The ADT will be conducted under the normal operating conditions for the autoclave with the exception that the wastes will be spiked with Agent VX on 10-gram (or smaller) pieces of the sample matrices.

5.4.1 Autoclave Operating Conditions

The optimization of feed rates is based on the ability to match the charge volumes and charge intervals to the temperature and steam supplied to the autoclave. Waste feed rate limits for the ADT are proposed at 259 ft³/charge. These feed rate limits are based on the capacity of the autoclave. Exact charge intervals and charge volumes will be finalized during the shakedown period. The charge volume will not exceed 259 ft³ and the total heat treatment time (i.e. time at temperature) will not be less than 3 hours at a minimum temperature of 275 °F. The DSHW will be notified if these charge intervals and charge volumes change during the shakedown period, but the charge volume and heat treatment time will not exceed the values listed in this plan and the minimum temperature will not be less than 275 °F.

The ADT will include triplicate sampling runs using four different waste matrices in each charge to the autoclave. These runs will be conducted to demonstrate removal of the agent from spiked samples to a level that has an agent concentration that is less than 1 % of the original agent mass spiked onto the sample matrix and an agent headspace concentration that is < 1.0 VSL. The ADT operating conditions will be discussed below.

The bins will be prepared such that air is drawn from the loading side to the hood at the mouth of the autoclave. The feed material will be DPE suit wastes in one bin, wood wastes in the second bin, LSS air hoses in the third bin, and the plastic drums in the fourth bin. The bins for each run will be prepared before the run begins. An alternative method of loading the wastes is to treat the wastes in the drums without transfer of the contents out of the drums. The shakedown will determine the optimum arrangement of the wastes for treatment.

Gas samples and condensate samples will be collected for each run. Samples of the gas exiting the condensers will be collected during the first two evacuations. Liquids condensed will be collected in a surge tank and sampled when this tank is pumped to the SDS tanks. It is anticipated that three condensate samples will be collected for each run.

The autoclave will operate with four bins at a time. The Test Director will authorize the run to begin when the bins have been placed in the autoclave and the door closed and locked. The autoclave will then be evacuated using the steam eductor to develop a vacuum on the system. During this time, the DAAMS on the duct between the condensers and the filter bank will sample for the presence of agent past the eductor. Once the vacuum goal is reached in the autoclave, the autoclave will have steam added and the heating of the wastes will begin. After the autoclave has heated to the minimum temperature and the minimum temperature has been maintained for 60 minutes, a vacuum will again be pulled on the autoclave and samples of the gas exiting the condensers will be collected by the DAAMS station. Steam will be added after the vacuum goal for the run has been reached and the steam will be used to reheat the wastes to the minimum temperature and then that temperature will be maintained for a minimum of 120 minutes. Adjustments to the time may be necessary based on system performance during shakedown. At the end of the heating cycle, a vacuum will again be drawn on the autoclave and once the vacuum is reached, the sample collections will be stopped and the autoclave will be

filled with ambient air. A blower will be used to add ambient air to the autoclave for a minimum of 60 minutes to cool the wastes.

After cooling, the air flow will be stopped to allow the agent concentration in the autoclave headspace to be determined. The autoclave will be allowed to reach a steady state agent concentration over a 30-minute period and then the ACAMS will be used to determine the agent concentration for two ACAMS cycles. If the agent concentration is below 1 VSL, then the waste will be removed and placed in a roll-off bin or in a DOT shipping container until shipped to a Subtitle C TSDF. If the agent concentration is ≥ 1.0 VSL, the waste will be treated in an addition cycle and this run will be declared an unsuccessful run and the samples will be discarded and the run designated a failed run. Another run will be made to replace the failed run. This process will be repeated until three successful runs have been completed.

5.4.2 Spiking Operations

Samples of DPE, LSS air hoses, and wood will be spiked with Agent VX to demonstrate the treatment of the wastes in the autoclave. After they are spiked, these samples will be placed in packages and sealed, placed in the wastes to be treated, then treated in the autoclave, recovered, and finally analyzed for Agent VX.

A 10-gram (or smaller) sample of the matrix will be prepared for spiking. The DPE sample will be a 10 gram (or smaller) piece cut from DPE suit material that may or may not have been used. Then, 1 μL of neat Agent VX will be placed in the center of the DPE sample. The LSS air hoses will have a 10-gram (or smaller) section cut from a length of hose and 1 μL of neat Agent VX placed on the interior portion of the hose. Particle board was chosen for the wood to be spiked, which will allow the Agent VX to be absorbed by the matrix. A 10-gram (or smaller) block of particle board will have 1 μL of neat Agent VX placed in the center of one surface.

Once the samples have been spiked, they will be placed in extraction thimbles and the thimbles placed in a sachet with a string attached to allow the sample to be recovered after autoclave treatment. The sachet and sample will be placed in a plastic bag to seal the sample so the agent cannot evaporate before it is placed in the wastes.

Three spiked samples will be placed in each waste bin, matching the spiked samples to the type of wastes in the bins. The spiked samples will have one placed near the center of the load, and the others will be placed in areas least likely to receive adequate treatment, thereby providing a worst case situation to demonstrate that the worst locations still receive adequate treatment. Provisions will be made to locate the sachets so they can be recovered for analyses.

The autoclave will be carefully unloaded to allow sachets recovery. Once the sachets are recovered, they will be placed in an amber glass bottle with a Teflon®-lined lid to protect the sample and minimize the evaporation of the agent or other compounds present. The bottle label will contain sample identification and identify the matrix and the sample location. The samples will be transferred to the CAL for Agent VX analyses.

5.5 AUTOCLAVE TEMPERATURE RANGES

The anticipated temperature limits for the autoclave are between 275 °F and 300 °F. The anticipated temperatures will vary within this range. Initial heating of the loaded bins will continue until all four thermocouples reach the minimum temperature of 275 °F. This temperature may be increased during shakedown depending on the autoclave performance, but the minimum temperature will not be less than 275 °F. The autoclave timer will continue the steam addition to maintain temperature for 60 minutes. A new vacuum will be drawn on the autoclave to lower the pressure to 10 inHg and then steam will be added to the autoclave to raise the waste temperature to a minimum of 275 °F again. This temperature will then be maintained for a minimum of 120 minutes, but this time may be extended based on system performance during shakedown. The bins will then cool down until they are removed from the autoclave.

5.6 ADT WASTE QUANTITIES

The autoclave has the capacity to hold four waste bins, which contain 64.7 ft³ each for a total volume of 258.8 ft³. For three runs, the total would be 776 ft³. The waste drums range in size from 30 gallons (gal) to 90 gal, so the number of drums emptied into the waste bin will vary based on the size of the drums. The volume of the waste load – rather than the weight – is proposed as an OPL because variations in mass do not affect the effectiveness of the autoclave treatment; i.e., the waste loads having different weights but the same volume will still be exposed to the same temperatures for the same period of process time; the process timer and thermocouples will ensure that these loads of various weights receive the same treatment time at the minimum temperature because the temperature of each waste load must reach the minimum temperature before the process timer begins to count down. The time it takes loads of waste to reach the minimum temperature will vary, with the heavier loads experiencing a longer “ramp-to-temperature” time than the lighter loads. The “ramp-to-temperature” time is not part of the treatment time.

5.7 AUXILLARY FUEL

Natural gas will be used to fire the boiler used to generate steam for the autoclave heating and the steam eductor. The natural gas used at TOCDF has an average heat content of 1043 Btu/ft³, and is 95.6 volume % methane, 2.5 volume % ethane, 0.41 volume % nitrogen, and 0.52 volume % carbon dioxide based on data from June 2005.

5.8 ADT REPORT

The final report will be a summary of the operating conditions recorded by the PLC, Agent VX concentrations in the autoclave headspace, condensate sample analyses, and copies of the final analytical data packages. The final report will be submitted within 90 days after the completion of the ADT to comply with the requirements for reports as stated in Title 40, Code of Federal Regulations, Part 63.1207(j) [40 CFR 63.1207 (j)].

5.9 AUTOCLAVE PERFORMANCE

The autoclave performance is discussed in this section as required by 40 CFR 264.601. The TOCDF believes that the conditions specified in Section 5.3 for the ADT will be adequate to meet the performance standards of 40 CFR 264.602 as incorporated into the TOCDF RCRA Permit while processing secondary wastes because:

- Testing conducted on a bench scale and the pilot plant scale indicates that the autoclave will be successful in the treatment of agent contaminated secondary waste (4, 5).
- The range of operating conditions planned for the ADT is within the design envelope.
- The autoclave is controlled by a PLC and whenever hazardous waste is being treated in the autoclave, the autoclave will not be opened until the headspace gas has been tested and determined to be safe.
- Each batch of waste treated will be monitored with an ACAMS to ensure it is safe to open the autoclave.
- One condition with three runs was selected to demonstrate the autoclave operation with secondary wastes.

6.0 SHAKEDOWN PERIOD PROCEDURES

Shakedown testing will proceed in accordance with the TOCDF ADT Shakedown Plan (see Appendix B). This shakedown plan defines all activities, methodologies, shakedown criteria, and compliance actions associated with the testing of the system. As stated in the shakedown plan, operating conditions will be maintained within the envelope of anticipated final operating limits throughout the shakedown period. These limits on operating conditions are based on good engineering practice, the bench-scale testing conducted at Southwest Research Institute (6), and the pilot-scale testing conducted at Bondtech's facility in North Carolina (5). These conditions will comply with the requirements of 40 CFR 264.601. Proposed operating conditions are preliminary, and final values will be confirmed or modified as shakedown progresses. Agent-contaminated secondary wastes will not be treated in the system at any time unless the conditions discussed above are satisfied.

This plan provides DSHW with a 60-day notice of the intent to conduct the ADT Shakedown and the anticipated start date. The shakedown will consist of up to 720 hours of secondary waste processing. The DSHW will be notified of the start date of the ADT two weeks before the test begins.

Once the approval of this plan is received from the DSHW, shakedown will commence as described in the ADT Shakedown Plan found in Appendix B. The entire system will be thoroughly tested during the shakedown period to verify that it performs in a safe, consistent, and predictable manner when processing secondary wastes.

6.1 ADT SHAKEDOWN

The objectives of the ADT shakedown are to:

- Demonstrate the ability of the autoclave to treat a variety of secondary wastes to agent concentrations that allow the safe handling of the wastes until they can be shipped to a Subtitle C TSDF based on feeding DPE suits, LSS hoses, and wood.
- Familiarize the operators and support personnel with the autoclave for decontaminating secondary waste.
- Determine the optimum charge volume and charge intervals for secondary wastes to be processed during the ADT.

- Establish operating conditions governing the treatment of secondary wastes in the autoclave that establish an operating envelope allowing the treatment of the secondary wastes based on waste temperatures and treatment times.
- Characterize the condensate samples to establish the final treatment for the condensate waste stream.
- Establish the process to monitor agent inside the autoclave to ensure satisfactory treatment of secondary waste.
- Demonstrate that the autoclave can safely and efficiently treat secondary wastes at the ADT feed rates.

The TOCDF will track ADT shakedown processing time as determined by the time the autoclave door seals to the time the door opens to remove the treated secondary wastes. Shakedown Time will be counted only when contaminated wastes are treated. Treatment of non-contaminated materials will not count against the Shakedown time.

The shakedown will involve a series of tests as described in the ADT Shakedown Plan (see Appendix B). During this time, secondary-waste-containing bins of increasing waste volumes will be charged to the autoclave. The values of the regulated operating parameters will be recorded before the charge volumes are increased or the charge interval adjusted. The charge volumes and charge intervals to be used during the demonstration test will be determined during the shakedown period. The TOCDF may request final modifications to the ADT Plan based on data obtained during the shakedown period. If changes to the ADT plan are necessary, TOCDF will coordinate them with the DSHW.

6.2 POST ADT OPERATION

Treatment of secondary wastes in the autoclave will continue after completion of the ADT, but the treated wastes will be held on-site until the preliminary data from the testing can be submitted to DSHW and approved. The preliminary data submitted will include the operational data from the autoclave, ACAMS data from the autoclave headspace, the analyses results from the spiked samples, and the agent results from the condensate samples. Upon approval of the preliminary data submittal, the wastes treated during the ADT and the post-test wastes treated will be sent to a Subtitle C TSDF. The headspace in the autoclave at the end of the treatment cycle will continue to be monitored during this time period to ensure the wastes have met their treatment conditions.

6.3 AUTOCLAVE PERFORMANCE

TOCDF believes that the conditions specified in Section 6.0 for the startup, shakedown, demonstration test, and post-test operation will be adequate to meet the performance standards of 40 CFR 264.602 while processing secondary waste because:

- Test results from the SWRI Report (6) indicate the agent is destroyed in the time frame used in processing secondary wastes in the autoclave.
- The headspace in the autoclave will be monitored for agent before the door is opened. If agent remains, the treatment time will be increased until the agent is destroyed.
- The autoclave operation during the shakedown and post-test periods will be controlled by the PLC and the SOPs directing the operation of the autoclave.

7.0 FINAL OPERATING LIMITS

The demonstration that agent is destroyed by treatment in the autoclave will be used to establish the operating permit limits for the autoclave. The successful completion of this ADT will establish the operating permit. The anticipated final operating conditions resulting from this ADT will be summarized in the ADT Report.

Agent destruction in the autoclave takes place by a hydrolysis reaction, which requires the water and temperature the steam introduces into the autoclave to go to completion. The temperatures measured in the wastes demonstrate operating conditions that ensure the destruction of agent. The waste feed rates demonstrated during this ADT and Agent VX will present the maximum challenge for the autoclave.

The following Operating Parameter Limits will be established by the ADT:

- Maximum Secondary Waste Feed Rates – The ADT will be performed as close to the maximum feed rates as possible. The final approved permit limit for waste feed will be the demonstrated ADT feed rates. The demonstrated rate will be a sum of the secondary wastes in the four bins or trays.
- Minimum Autoclave Temperature – The minimum autoclave temperature will be demonstrated during the ADT, provided that the agent is destroyed.
- Agent Concentrations – An agent concentration in the headspace below the detection limit in the autoclave will be demonstrated by the ADT. This demonstration will allow TOCDF to process the secondary wastes contained in the bins.
- Minimum Vacuum Differential Pressure – The final approved permit limit for minimum vacuum pressure in the autoclave will be determined during the ADT provided acceptable agent concentrations are demonstrated.

8.0 REFERENCES

- (1) ***Test Methods for Evaluating Solid Waste, Physical/Chemical Methods***, 3rd Edition, including Update III, USEPA, SW-846, December 1996.
- (2) Muro, N.B., S.S. Talmage, G.D. Griffin, L.C. Waters, A.P. Watson, J.F. King, V. Hauschild, "The Sources, Fate, and Toxicity of Chemical Warfare Agent Degradation Products," ***Environmental Health Perspectives***, 1999, 107, No. 12, December, 933-974.
- (3) Yang, Y.C., J.A. Baker, J.R. Ward, "Decontamination of Chemical Warfare Agents," ***Chem. Rev.***, 1992, 92, 1729-1743.
- (4) ***Chemical Agent Data Sheets***, EO-SR-74001, December 1974.
- (5) ***Autoclave Evaluation Test Report***, Continental Research and Engineering. LLC, Englewood, CO, Revision 0, April 21, 2008.
- (6) J. Scott, R. Martinez, ***Autoclave Secondary Waste VX on DPE and Wood***, Interim Report, Southwest Research Institute, San Antonio, TX, September 30, 2008.
- (7) ***Guidance on Setting Permit Conditions and Reporting Trial Burn Results***, EPA/625/6-89/019, January 1989.
- (8) **Attachment 22 to the TOCDF Permit, Agent Monitoring Plan**, EG&G Defense Materials, Inc., TOCDF CDRL 23.
- (9) "Standard Practices for Sampling Water from Closed Conduits," ***ASTM D 3370-95a*** (Reapproved 1999), ASTM International, West Conshohocken, Pennsylvania.